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SPECIAL REPORT

Simple Field Screening Method for White Phosphorus (P_4) in Sediment

Marianne E. Walsh, Charles H. Racine, Charles M. Collins,
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October 1995

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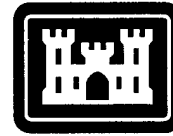
Abstract

A simple field screening method to detect white phosphorus particles in sediment is described. A thin layer of wet sediment is heated until all water evaporates. The presence of white phosphorus is indicated by visual detection of the inflammation of white phosphorus particles that occurs at relatively low temperatures (less than 40°C) once a protective layer of water is removed. The field screening method consistently gave positive results for samples where solvent extraction followed by gas chromatography indicated white phosphorus concentrations above 1 µg/g. A more sophisticated method, based on solid-phase microextraction and gas chromatography determination, was also tested. Concentrations less than 1 µg/kg were detectable.

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**US Army Corps
of Engineers**

Cold Regions Research &
Engineering Laboratory

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PREFACE

This report was prepared by Marianne E. Walsh, Chemical Engineer, Applied Research Division, Charles H. Racine, Research Ecologist, Charles M. Collins, Physical Scientist, and Philip Thorne, Physical Scientist, Geological Sciences Division, Research and Engineering Directorate, U.S. Army Cold Regions Research and Engineering Laboratory, Hanover, New Hampshire, and Carl Bouwkamp, Biologist, U.S. Army Environmental Hygiene Agency, Aberdeen Proving Ground, Maryland.

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Simple Field Screening Method for White Phosphorus (P₄) In Sediment

MARIANNE E. WALSH, CHARLES H. RACINE, CHARLES M. COLLINS,
CARL BOUWKAMP AND PHILIP G. THORNE

INTRODUCTION

Analytical methods have been developed for white phosphorus (WP, P₄) residues in sediment and water (Walsh and Taylor 1993 and Walsh 1995). These methods require that a field sample be brought to a laboratory where a subsample is extracted with solvent and the extract analyzed by gas chromatography. The cost of this analysis is approximately \$120 per sample. These methods have been used to analyze several thousand samples from Eagle River Flats (ERF), the impact area on Fort Richardson, Alaska (Racine et al. 1992, 1993) and from 24 other Army sites (Simmers et al. 1994).

WP contamination coincides with areas of numerous impact craters with standing water (Racine et al. 1993). Within these areas, high concentrations of WP are distributed in areas less than a meter in diameter, which presumably correspond to the point of impact of a WP projectile. Immediately surrounding the impact points are large areas where WP is at very low concentration (<0.001 µg/g) (Walsh and Collins 1993). Samples with high concentrations were found to contain WP particles ranging in size from less than 0.1 to 5 mm (Racine et al. 1993, Walsh and Collins 1993). Since Army training areas tend to be large (several square kilometers) and WP tends to be heterogeneously distributed in discrete and relatively small (less than 1-m²) areas, the number of samples required to screen an area for contamination can be unrealistically large. Depending on the objectives of a sampling effort, the costs and time required for laboratory analyses may be unacceptable.

The objective of this work was to develop a quick and easy, low cost method to detect milli-

meter-size particles of white phosphorus. The method was designed so that many samples could be tested for white phosphorus in the field without the use of organic solvents or sophisticated equipment. In addition, a screening procedure based on solid phase micro-extraction (SPME) (Zhang and Pawliszyn 1993) was tested. This SPME procedure, however, requires the use of a field-portable gas chromatograph.

METHODS

Materials

White phosphorus (P₄) and isooctane were obtained from Aldrich Chemical Company.

In the laboratory, white phosphorus particles with diameters ranging from 0.3 to 1.8 mm were produced under water from molten white phosphorus using a Gilson Microman Positive Displacement Pipet (size M25) equipped with disposable capillaries and pistons. The diameter of each particle was measured using a SPI (Swiss Precision Instruments) 6-in. caliper with 0.1-mm graduations.

Solid phase micro-extraction fiber (100-µm polydimethylsiloxane) assemblies were obtained from Supelco.

Collection of sediment samples

Sediment samples were collected from Eagle River Flats, Ft. Richardson, Alaska, a site contaminated from white phosphorus munitions (Racine et al. 1992, 1993). Sediment samples were collected by two methods. For the first method, a sample site was chosen, and then several small samples of the surface sediment within a 0.5-m radius

were combined in a 500-mL jar. Each jar was filled to capacity. The second sampling method involved scooping several samples of sediment into a wash bucket equipped with a no. 30-mesh (0.59-mm) sieve and stirring the sample underwater. This procedure preconcentrated the sample by removing most of the fine-grained silt particles. The material left on the sieve was placed in a 500-mL jar with enough water to cover the sample. Samples collected by either method were cooled to 4°C and stored in the dark until analyzed.

Detection of WP particles

Field method

Each sample was prepared for analysis by taking an approximately 20- to 30-mL subsample and spreading it across the bottom of a 20-cm-diam. aluminum pie pan. The subsample was spread in a thin layer (approx. 1 mm thick). Highly organic, fibrous samples were pulled apart.



a. Preparation of samples for field test.



b. A thin smear of sediment is heated until all water evaporates.

Figure 1. Field test for the detection of white phosphorus particles in sediment.

Depending on the size of the sample and the objective of the analysis, several subsamples were taken.

To detect WP particles, the aluminum pie pan with a thin smear of sample was placed on a heated surface. In the field, we used a double burner propane camp stove (American Camper propane stove) and a cast iron pancake griddle (Wagner's 1891 cast iron cookware) (Fig. 1a and 1b). In the lab, we used a hot plate (Corning) set on the highest setting. The hot plate was placed in the back of the fume hood and the shield of the fume hood pulled down.

The heat from the stove or hot plate evaporated the water from the sample and ignited WP particles, if they were present. A positive test for a WP particle was indicated by a localized area of intense smoke and flame and the formation of a bright orange residue (Fig. 2). The orange residue, a mixture of oxidation products of WP (Daasch et al. 1969), is hygroscopic and, if the residue was surrounded by sediment, the sediment also appeared moist. The moist sediment was darker than the light gray, dried ERF sediment, forming a dark halo around the orange residue. The orange residue also had a garlic-like odor, probably due to the production of P_4O_6 .

Solid phase micro-extraction screening method

A 40-g wet sediment subsample, measured with a field-portable balance (Ohaus model CT200), was placed in a 120-mL jar equipped with a septa-top. A 10-mL aliquot of reagent grade water (MilliQ, Millipore) was added and the sample was equilibrated at room temperature for one hour.

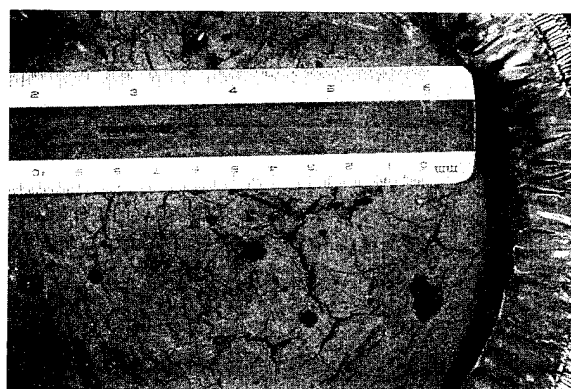


Figure 2. Positive test for a WP particle. This consists of a localized area of intense smoke and flame, and the formation of a bright orange residue (dark gray in this black-and-white picture).

The sample was shaken vigorously by hand; then the SPME phase was exposed to the headspace for 5 min. The SPME phase was immediately transferred to a heated (200°C) injection port of a portable gas chromatograph (SRI Model 8610) equipped with a nitrogen-phosphorus detector. The polydimethylsiloxane fused-silica column (J&W DB-1, 15 m × 0.53 mm i.d., 3-μm film thickness) was maintained at 80°C and the carrier gas was nitrogen set at 30 mL/min.

Laboratory method

White phosphorus concentration (μg/g) was determined using isooctane extraction of a 40-g wet subsample followed by gas chromatography (Walsh and Taylor 1993).

RESULTS AND DISCUSSION

Initial tests

Initially, we collected samples from sites where, using the laboratory method, we previously had detected WP and from two sites where WP was not detected (Racine et al. 1993a and b). At each sample site, a 500-mL jar was filled with sediment and subsamples were smeared across the bottom of aluminum pie pans. Samples were tested for WP by heating each pan on a camp stove.

For each sample where we had detected WP above 1 μg/g by the laboratory method, we obtained a positive result by the field test for at least

one subsample (Table 1). Samples with lower concentrations apparently did not contain white phosphorus particles large enough to produce a visible flame or leave orange residue. Based on these results, we concluded that the field test provided a means to quickly detect large (millimeter size) WP particles if they were present in the subsample taken, but several subsamples may be required to obtain a positive result for samples with few particles.

We then questioned how many subsamples should be tested if the first subsample yielded a negative result. A 500-mL jar contains sufficient material for approximately 20 subsamples; however, testing so many subsamples per sample would be extremely tedious. Next we tried pre-concentrating what remained of samples 2–8 (Table 1) by sieving through a 30-mesh (0.59-mm) sieve to remove most of the fine-grain silts and clays and reduce the volume of the sample so that only one test need be run. No more particles were found in samples 2–6; however, four more particles were found in sample 7 and two more in sample 8.

Composite sampling

Since sieving provides a means to reduce the volume of a sample, we tested sieving in the field as a means to create composite samples from a large area (Fig. 3). Dabbling ducks at Eagle River Flats have proved to be efficient samplers of white phosphorus particles as evidenced by their high mortality. We reasoned that by simulating the way ducks feed, by sieving several small sediment samples over a large area, we might increase the efficiency of sampling. Our concern over a sampling method stems from the way in which the marsh was contaminated. Projectiles containing white phosphorus produce discrete and relatively small areas of contamination. For example, following the detonation of 81-mm mortar rounds the areas containing the majority of the white phosphorus residue were only 0.5 m in diameter (0.2 m²) (Walsh and Collins 1993). To sample for such a small hot spot with 90% confidence ($\beta = 0.10$) using a grid pattern of sampling would require a 0.9-m grid spacing (Gilbert 1987). When we consider that Eagle River Flats contains 700,000 m² of ponds, the grid approach to sampling would be unrealistically costly, even where multiple hot spots exist.

Table 1. Comparison of white phosphorus concentrations found by laboratory method and number of white phosphorus particles detected by field method.

Sample	Sample ID	WP conc. (μg/g)	Number subsamples tested	Number WP particles found
1	1248	ND	2	0
2	AEHA BT1	ND	10	0
3	AEHA PB1	0.0143	10	0
4	AEHA D2	0.079	10	0
5	AEHA DUP1	0.205	10	2
6	AEHA D1	0.43	10	0
7	AEHA C2	1.6	10	1
8	240	2.32	10	1
9	53	10.2	2	1
10	1247	88	1	23
11	1245	168	2	7
12	110	590	2	2
13	AEHA PB2	1740	1	12
14	1246	3,071	1	68
15	MHB site	5600	1	>100

ND = not detected

To test the compositing approach to sampling, two people took samples simultaneously in ponded areas of Eagle River Flats. The first person chose a site and then took a 500-mL sediment sample by combining several small samples of the surface sediment within a 0.5-m radius. The second person used a wash bucket equipped with



Figure 3. Composite samples collected using a large, long-handled stainless steel spoon and wash bucket equipped with a no. 30-mesh (0.59-mm) sieve. The composite was made from several sediment samples over a radius of up to 5 m or along a transect which included two or more discrete sample sites.

a no. 30-mesh (0.59-mm) sieve and a large, long-handled stainless steel spoon to collect several sediment samples over a radius of up to 5 m or along a transect that included two or more discrete sample sites (Fig. 3). The wash bucket was held underwater and the sample continuously stirred to reduce the volume. Samples were taken until the volume of material on the sieve was approximately 500 mL.

Sieved samples were tested for the presence of WP particles by heating at least four subsamples in the field. The test was performed by a field technician with no prior experience using this test. The technician was instructed to examine each plate for orange residue (Fig. 4). Then both sieved and discrete samples were returned to the laboratory where a subsample was extracted with isooctane and analyzed by gas chromatography.

Of the 17 samples taken, nine were blank by all three analyses (Table 2). One sample (no. 12) was reported to be positive by the field test, but not by the laboratory method. This result may be due to heterogeneity in the distribution of particles or by flecks of iron in the sample that resemble the orange burn residue left by a white phosphorus particle. The remaining seven samples were positive by the field test and by the laboratory analy-

Table 2. Number of white phosphorus particles detected in sieved composite samples by the field method and WP concentration found by the laboratory method in a separate subsample of each sieved composite.

	Sites	Number of subsamples from sieved composite	Number of WP particles detected in sieved composite	WP concentration* ($\mu\text{g/g}$)	
				Sieved composite	Not sieved discrete
1	1295-1296	4	1	0.200	ND
2	1297	6	1	0.034	0.017
3	1292	4	60	603	0.03
4	1289	4	0	0.012	0.068
5	1293	4	59	923	0.34
6	1299-1300	5	2	26.0	0.42, 0.001
7	1290	4	46	1410	431
8	1301-1306	4	0	ND	ND
9	1310	4	0	ND	ND
10	1313	4	0	ND	ND
11	1314	4	0	ND	ND
12	1317	4	3	ND	ND
13	1318	4	0	ND	ND
14	1319	4	0	ND	ND
15	1321	4	0	ND	ND
16	1322	4	0	ND	ND
17	1324-26	4	0	ND	ND

* Determined by laboratory method.

ND = not detected

Note: Also shown are WP concentrations found in discrete samples taken from the same location as the sieved composites.



Figure 4. Technician in field examining samples for evidence of WP particles.

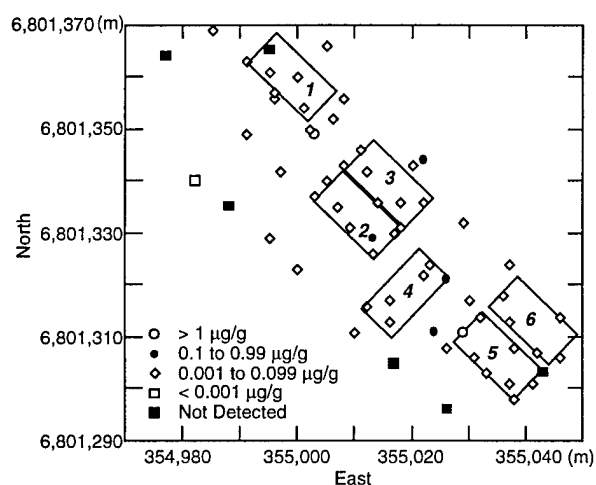


Figure 5. Map showing that WP concentrations and boundaries of six 7.5-m x 20-m areas where composite samples were taken and analyzed by the field test.

sis of the sieved composite. WP concentrations in the sieved composite, as determined by the laboratory method, were higher in six out of the seven samples by up to four orders of magnitude than the discrete sample (Table 2). The higher concentration was caused by preconcentration of white phosphorus particles or by the increased likelihood of hitting a hot spot when taking samples over a large area.

Additional composite samples were taken from a ponded area in Eagle River Flats that had been intensively sampled in the past and where many ducks were observed to die of white phosphorus poisoning. The area was divided into six 7.5-m x 20-m blocks and 25 samples were collected from each block (Fig. 5). These samples were mixed together and sieved to produce a single sample

for each block. Each composited sample was tested using the field method by dividing into several pans and heating. (One drawback of sieving is that it also concentrates organic matter that makes it difficult to spread the sample across a pie plate in a thin layer.) After removing as much material as possible from each sample jar for the field tests, 10.0 mL of isooctane was added to each jar to rinse the sides and the rinsate analyzed by gas chromatography.

White phosphorus particles were detected in two of the six areas, four in block 5 and one in block 2. The diameters of the orange spots produced during these tests were measured, ranging from 1.7 to 3.4 mm for the sample from block 5 and 4.9 mm for the sample from block 2. WP was also detectable in the rinsate from the block 5 and block 2 jars and not in the rinsate from the other jars. Block 5 was the only area where a WP concentration above 1 µg/g was detected previously.

Estimation of WP particle size distribution

This field technique is not quantitative, but it may yield some information on the WP particle size distribution in a subsample. While testing sediment samples using the field method, we noticed that the diameter of the orange residue varied from a fraction of a millimeter to over a centimeter. To see if there was a relationship between the size of a WP particle and the diameter of burn residue, in the laboratory we produced spherical WP particles ranging in diameter from 0.3 to 1.8 mm and placed the particles in a smear of wet sediment in an aluminum pie pan. We then heated the sample until the water evaporated and the WP particles ignited. We measured the diameter of the orange residue produced, and found good correlation ($r^2 = 0.903$) (Fig. 6) between the diameter of the residue and the diameter of the original particle.

Interferences

In Eagle River Flats sediments, an orange residue may be left by iron fragments and by some invertebrates that live in the sediments. For example, the burned remains of the midge larvae *Chironomus* sp., commonly called blood worms, were mistaken for the orange residue left by WP particles. For a test to be considered positive for white phosphorus, the observations should require both a flame and the formation of orange residue. Adherence to these criteria should eliminate most false positives.

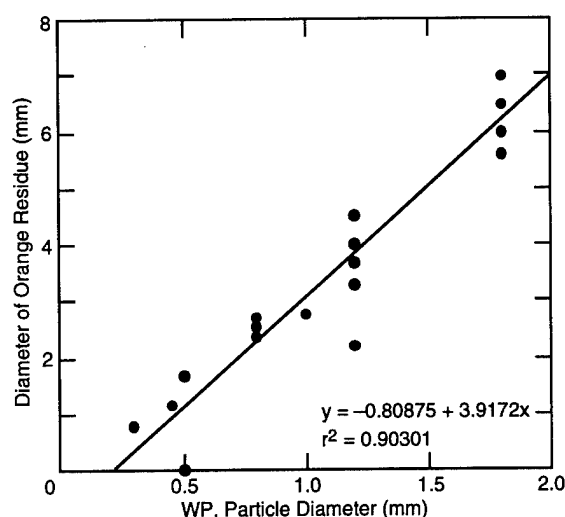


Figure 6. Diameters of orange residue left by WP particles of known diameter after placement in a smear of mud on an aluminum pie plate and heating until water evaporates.

The orange residue produced from WP is acidic and rich in orthophosphate. However, the sediment at ERF is highly buffered and is also rich in orthophosphate. Therefore efforts were unsuccessful to confirm a positive field test measuring pH or orthophosphate.

SPME and future work

The field screening approach described in this report is simple, quick and unsophisticated. Positive results were obtained for samples where the laboratory method indicated WP concentrations above 1 $\mu\text{g/g}$. We next tested a SPME procedure (Fig. 7) to allow detection of lower concentrations of WP without solvent extraction. This procedure does require the use of a gas chromatograph; therefore, field personnel would need to be trained in GC setup and maintenance. A series of discrete sediment samples were collected from Eagle River Flats. For each sample, a 40-g wet subsample and 10 mL of reagent grade water were placed in a 120-mL jar equipped with a septum cap. The jars were sealed and manually shaken 10 times so that the sample was well mixed and coated the sides of the jar. Samples stood at room temperature (20°C) for one hour, and then each sample was analyzed as follows. The jar was shaken an additional 10 times, and the SPME fiber was exposed to the headspace for 5 min. Immediately following exposure to the headspace, the SPME fiber was inserted into the injection port of the gas chromatograph, and a chromatogram ob-

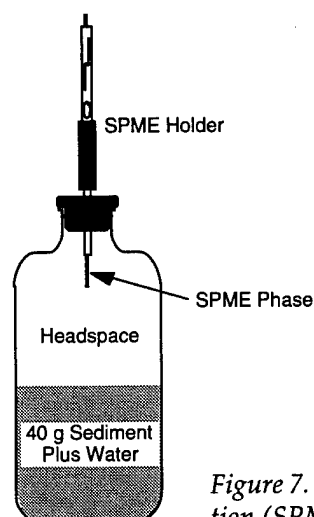


Figure 7. Solid phase micro-extraction (SPME) of a sediment sample to test for the presence of white phosphorus. Following exposure to the headspace above the sample, the SPME phase is transferred directly to the injection port of the gas chromatograph.

tained. Following analysis by headspace SPME, a 10-mL aliquot of isooctane was added to each sample jar, and the samples shaken for 18 hours. A 1- μL aliquot of the isooctane extract was injected into the gas chromatograph. The mass of white phosphorus detected per injection by each method was calculated based on external calibration standards. The mass found in the 1 μL of isooctane was also used to calculate the concentration in $\mu\text{g/g}$ (Table 3). For the three samples where the white phosphorus concentration was greater than 0.5 $\mu\text{g/g}$, two additional 40-g subsamples were spread across the bottom of an aluminum pie plate and heated.

The amount of WP detected in the headspace of each sample by SPME correlated well with that found by solvent extraction. Of the 19 samples tested, 13 were negative by both methods. For the six positive samples, the amount of WP detected by SPME was proportional to that found by solvent extraction, i.e., the highest masses of WP detected were for those samples with the highest concentration. Both methods were comparable in detection capability. The certified reporting limit (CRL) of the solvent extraction procedure is 0.00088 $\mu\text{g/g}$. The lowest concentration detected in the positive samples was slightly greater than the CRL (0.00094 $\mu\text{g/g}$), and SPME also gave a positive result for this sample. Future work on the SPME procedure will be to attempt to calibrate for quantitative results.

For those samples that were subjected to the

Table 3. WP detected by solvent extraction, SPME and field test.

Sample* ID	WP conc. ($\mu\text{g/g}$) [†]	WP mass (pg) detected		Hot plate field test	
		Isooctane**	SPME	Number of particles detected	Diameter of residue (mm)
BT-CC-1a	0.00094	3.8	6.0		
old 248	0.0015	6.0	3.6		
old 250	0.031	120	140		
CP-CC-02 ^{††}	0.55	2,200	>5,400	0	
MHB site ^{††}	9.52	38,000	>11,000	7	0.4-3.7
CP-CC-01 ^{††}	70.1	280,000	>11,000	1	3.5
CP-CC-03	<0.00088	ND	ND		
CP-CC-04	<0.00088	ND	ND		
CP-CC-05	<0.00088	ND	ND		
CP-CC-06	<0.00088	ND	ND		
CP-CC-07	<0.00088	ND	ND		
CP-CC-08	<0.00088	ND	ND		
CP-CC-09	<0.00088	ND	ND		
MHR site	<0.00088	ND	ND		
MW site 1	<0.00088	ND	ND		
MW site 2	<0.00088	ND	ND		
MW site 3	<0.00088	ND	ND		
MW site 4	<0.00088	ND	ND		
MW site 5	<0.00088	ND	ND		

* Same sample used for isooctane extraction and SPME.

[†] WP concentration determined by isooctane (10 mL) extraction of 40-g sediment sample.

** WP mass detected in 1- μL aliquot of 10-mL extract.

^{††} Samples were overrange. Isooctane extracts diluted to within linear range.

ND = Not detected

Table 4. Estimation of WP concentration based on the number and diameter of WP particles detected by field method.

Sample	Diameter of residue (mm)	Estimated* WP particle diameter (mm)	Estimated [†] mass of WP particle (mg)	Estimated** WP conc. ($\mu\text{g/g}$)
CP-CC-01	3.5	1.1	1.3	16.0
MHB site	3.7	1.2	1.5	
	1.6	0.6	0.2	
	1.5	0.6	0.2	
	1	0.5	0.09	
	0.6	0.4	0.04	
	0.6	0.4	0.04	
	0.4	0.3	0.03	
Sum for MHB site = 2.1				26.1

* Assuming each particle is a sphere.

[†] Density of WP is 1.82 g/mL

** Based on 80 g of sediment (two 40-g subsamples).

hot plate field test, WP particles were detected in two out of the three samples (Tables 3 and 4). The diameter of the residue left by each particle was measured, and the equation presented in Figure 6 used to estimate the diameter of each WP particle prior to burning. The total mass of the WP was estimated based on the density of WP (1.82 g/mL)

and the assumption that each particle was spherical. WP concentrations for the samples where comparison was possible were estimated and found to be of the same order of magnitude as that obtained by the laboratory method. Since the presence or absence of one millimeter-size particle in a subsample would completely change the con-

centration estimate, we can never expect excellent quantitative agreement.

Each screening method has certain advantages. The hot plate field test requires minimal training and equipment, but detection capability is limited to samples containing millimeter-size particles of white phosphorus. Also, for some sites where the identity of contaminants is unknown, heating a sample might not be prudent in terms of safety. The SPME approach provides much greater sensitivity than the hotplate method, but requires a gas chromatograph and an analyst trained in GC operation and maintenance.

CONCLUSIONS

A simple field-screening method to detect white phosphorus particles is described. The method is based on the visual detection of the inflammation of white phosphorus particles at relatively low temperatures (less than 40°C) once a protective layer of water is removed. The field screening method consistently gave positive results for samples where the laboratory method indicated WP concentrations above 1 µg/g. It also gives some idea of the size and number of particles. The number of false positives were few if the criteria for a test to be considered positive are the observations of 1) a localized area of intense smoke and/or flame and 2) the formation of orange residue.

A more sophisticated technique, based on solid-phase microextraction and GC determination, was tested and found to be comparable in detection capability to the laboratory method.

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13. ABSTRACT (Maximum 200 words) A simple field screening method to detect white phosphorus particles in sediment is described. A thin layer of wet sediment is heated until all water evaporates. The presence of white phosphorus is indicated by visual detection of the inflammation of white phosphorus particles that occurs at relatively low temperatures (less than 40°C) once a protective layer of water is removed. The field screening method consistently gave positive results for samples where solvent extraction followed by gas chromatography indicated white phosphorus concentrations above 1 µg/g. A more sophisticated method, based on solid-phase microextraction and gas chromatography determination, was also tested. Concentrations less than 1 µg/kg were detectable.					
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